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# EUROPEAN PATENT APPLICATION

21 Application number: 83106262.5

51 Int. Cl.<sup>3</sup>: A 23 L 1/236

22 Date of filing: 27.06.83

## BEST AVAILABLE COPY

30 Priority: 28.06.82 JP 111304/82  
15.02.83 JP 23459/83

43 Date of publication of application:  
11.01.84 Bulletin 84/2

64 Designated Contracting States:  
CH DE FR GB IT LI NL SE

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54 Stabilized aspartame composition, a process for its production and an aqueous food.

57 A stabilized aspartame composition comprising aspartame and a sugar derivative selected from the group consisting of a cyclodextrin and a sucrose fatty acid ester is described along with methods of producing such compositions, either as aqueous or solid forms suitable for use as table sweeteners or as foods containing aspartame.

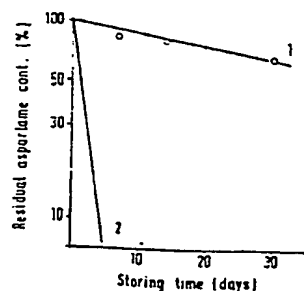


FIG. 1

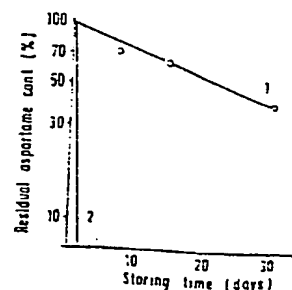


FIG. 2

TITLE OF THE INVENTION

STABILIZED ASPARTAME COMPOSITION, A PROCESS FOR  
ITS PRODUCTION AND AN AQUEOUS FOOD

BACKGROUND OF THE INVENTION5 Field of the Invention:

The present invention relates to stabilized compositions of aspartame such as aqueous foods containing aspartame which contain a stabilizing agent which allows long-term storage of the composition or  
10 food without significant deterioration of the aspartame.

Description of the Prior Art:

With the recent increase in the consumption of low-calorie beverages and health foods, aspartame, a  
15 low-calorie sweetener offering an intense sweetness of very good quality, has come to be considered one of the best candidates for incorporation in such beverages and foods. But as is often pointed out, one great problem with storing aspartame ( $\alpha$ -L-aspartyl-L-phenylalanine  
20 methyl ester) in the presence of water is that its quality is impaired during storage due to the pH and temperature at which such foods and beverages are normally stored. In foods and beverages wherein both water and aspartame are present under weakly acidic or  
25 alkaline conditions, in foods and beverages containing

aspartame together with a high water content  
irrespective of pH, or in foods and beverages which are  
manufactured, stored or distributed at high to medium  
temperatures while containing aspartame in the presence  
5 of water, the aspartame is partially decomposed to  
diketopiperazine, which is non-toxic and perfectly safe  
but has no sweetness. This results in reduced  
sweetness of the food or beverage containing the  
aspartame. This decrease in the quality of aspartame  
10 in the presence of water during storage can be a  
barrier to its extensive use in sweet aqueous foods and  
beverages, such as soft drinks and juices, that  
currently use high volumes of sweeteners and  
potentially are a major market for low-calorie  
15 sweeteners. Therefore, enhancing the storage stability  
of aspartame in water is very important for expanding  
its use in processed foods.

This demand has been partially met by storing  
aspartame at low temperatures and at a controlled pH  
20 (the aspartame is stable in a pH range of 4 to 4.5), by  
adding a Ca or Mg ion releasing material (Japanese  
Patent Application (OPI) No. 77569/75), or by encapsu-  
lating the aspartame (Japanese Patent Application (OPI)  
No. 202268/82 and Japanese Patent Publication No.  
25 53063/82). However, none of these techniques are  
completely satisfactory since they cannot be applied to  
soft drinks and juices with sufficient ability to  
stabilize the aspartame contained therein.

Furthermore, aspartame does not have sufficiently high dispersibility or solubility in water to be used as a table sweetener or an additive to processed foods as easily as sugar. One method conventionally  
5 used to improve the solubility properties of aspartame is to dry it together with an excipient highly soluble in water. The use of an excipient contributes to the improvement of the dispersibility and solubility of the aspartame, but in many cases, aspartame treated by this  
10 method does not become as stable in water as aspartame that is coated with water-insoluble substances such as fats and oils. Therefore, no conventional technique is ideal in that it cannot satisfy simultaneously the requirements of high storage stability and improved  
15 solubility and dispersibility which are generally incompatible with each other.

#### SUMMARY OF THE INVENTION

It is an object of the present invention to provide a composition containing aspartame which is  
20 stable toward long-term storage in an aqueous environment.

It is a further object of this invention to provide aspartame-containing foods and beverages having a high water content which are stable during long-term  
25 storage.

It is yet another object of this invention to provide a method of producing compositions of the invention and foods and beverages of the invention which are stable during long-term storage.

5        These and other objects of the invention as will hereinafter become more readily apparent have been accomplished by providing a stabilized aspartame composition comprising aspartame and a sugar derivative selected from the group consisting of cyclodextrins and  
10        sucrose fatty acid esters.

BRIEF DESCRIPTION OF THE DRAWINGS

A more complete appreciation of the invention and many of the attendant advantages thereof will be readily obtained as the invention becomes better  
15        understood by reference to the following detailed description when considered in connection with the accompanying drawings, wherein:

FIGURE 1 shows the long-term stability of aspartame in (1) aqueous solutions containing 1% of a  
20        sucrose fatty acid ester and (2) otherwise identical solutions containing no such ester; and

FIGURE 2 shows the long-term stability of aspartame in (1) aqueous solutions containing 3% of a  
25        sucrose fatty acid ester and (2) otherwise identical solutions containing no such ester.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

In the process of achieving the present invention, the present inventors made extensive studies to develop an aspartame composition which has improved stability in water as well as enhanced dispersibility and solubility and have found that the stability of aspartame in water can be improved by using it together with a cyclodextrin. The dispersibility and solubility of aspartame in water can also be enhanced by entrapping the aspartame in the cyclodextrin. Additional studies further demonstrated that the stability of aspartame in water can be further improved by incorporating a sucrose fatty acid ester conventionally used as an edible surfactant into the aspartame composition, either alone or in combination with a cyclodextrin.

Accordingly, one aspect of the present invention relates to an aspartame composition which contains a cyclodextrin preferably in a molar ratio of 200:1 to 1:50 with respect to the aspartame as well as to an aqueous food or beverage that contains said composition. The invention also relates to a process for producing the sweetener composition by mixing the aspartame with the cyclodextrin in the presence of water and/or an aqueous solvent, optionally followed by drying of the mixture.

The cyclodextrin used in the present invention (hereafter abbreviated as CD) is a cyclic

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oligosaccharide homolog that is also known as a  
cycloamylose. It consists of 6 to 10 D-glucopyranose  
groups bonded through  $\alpha$ -(1,4)-glucoside bonds to form a  
cyclic structure. It is named  $\alpha$ -CD,  $\beta$ -CD or  $\gamma$ -CD  
5 according to the degree of polymerization (6, 7 or 8  
glucose units). The interior of the ring contains C-H  
bonds or ether bonds and is hydrophobic, while the  
exterior of the ring is interspersed with OH groups and  
is highly hydrophilic. Because of this structure, CD  
10 is capable of entrapping various compounds in the  
interior of the cyclic molecule. Previously, CD has  
been used in foods in order to mask a fishy smell (in  
the protein processing industry), to prevent oxidation  
(in the fat and oil processing industry), to preserve  
15 flavor (in spices), and to prevent moisture absorption  
and deliquescence (in sugared foods).

CD is usually produced from starch by treating it  
with an amylase or a similar enzyme produced from  
Bacillus macerans or an alkali-resistant bacterium.  
20 There are no particular limitations on the CD that can  
be used in the present invention with respect to the  
conditions for producing it or other factors.  
According to a preferred embodiment of the present  
invention,  $\alpha$ -CD,  $\beta$ -CD and  $\gamma$ -CD may be used either  
25 independently or as a mixture, although the intended  
object of the present invention can be achieved with  
any type of CD.

The stability of aspartame in water can be improved by using CD in a trace amount in a molar ratio of 1:200. Using CD in an amount greater than 50 times the amount of aspartame is not only uneconomical, but  
5 it is also detrimental to the purpose of reducing the caloric content. Therefore, the practical acceptable molar ratio of aspartame to CD is in the range of from 200:1 to 1:50.

The stability of aspartame in water can be  
10 improved by simply using it together with the CD, but for the purpose of improving its dispersibility and solubility in water in addition to its stability in water, it is preferred that at least a part of the aspartame be entrapped in the CD. One particularly  
15 useful application of this aspect of the invention is that it allows the preparation of an aspartame sweetener composition; i.e., a composition intended for use by the ultimate consumer as a table sweetener. Thus the aspartame compositions containing a cyclo-  
15 dextrin, and particularly those in which aspartame is entrapped within the cyclodextrin, have sufficient solubility and dispersibility that they may be used as table sweeteners, either in solid or liquid form.

Any method can be used for entrapping the  
20 aspartame in the CD, and a typical method is described for purposes of illustration. First, the aspartame is mixed with CD in the presence of water or an aqueous



solvent such as aqueous alcohol. If desired, an excipient such as dextrin, sugar, sugar alcohol, polysaccharides, protein, amino acid, or salt or a flavor or any other quality modifier may also be

5 used. Water and other aqueous solvents may be used in any amount that enables the formation of the desired entrapped compound. However, if the mixture must be subsequently dried, water and other aqueous solvents should not be used in an amount greater than that

10 necessary for forming a slurry or paste of the mixture of aspartame and CD (e.g., 0.3 to 3 times the amount of CD) in order to avoid an increase in the energy cost and to prevent browning and the loss of the sweetness of the aspartame due to extended drying at elevated

15 temperatures. The resulting solution or slurry containing both the aspartame and CD is preferably heated to a temperature between about 30 and 90°C in order to increase the solubility of the aspartame and CD to such an extent that they can be dissolved in the

20 least possible amount of water (i.e., a uniform product is produced by using a minimum amount of water and the efficiency of the subsequent drying is increased). The aspartame may be added either as a mixture with CD or as a separately prepared solution or slurry of the

25 aspartame. But apart from the case where the aspartame has been made easily soluble by using another excipient, the preparation of a separate solution or

slurry of the aspartame is preferred in order to facilitate the subsequent step of dissolving the CD.

The slurry is intimately mixed in a homogenizer, a kneader or the like for a period which generally ranges from about 30 minutes to a few hours. Mixing continues until the viscosity of the slurry is slightly increased to provide a pasty hydrous product wherein all or part of the aspartame is entrapped in the CD. When the mixture of aspartame and CD is a solution, it is stirred for a period of about 30 minutes to a few hours. In consequence, the greater part of the entrapped aspartame is obtained as a precipitate, which may be recovered by any suitable method such as removal of the solvent or recrystallization by cooling. . . . Removal of the solvent is desirably effected by vacuum drying, by drying with dehumidified air, or by a similar method at low temperatures, preferably below 80°C.

Needless to say, the solution or slurry containing the aspartame and CD may be directly used as a liquid or pasty sweetener.

The dried product also can be distributed as a sweetener after it is formed into a suitable shape, such as a powder, granules, tablets, or cubes, either independently or together with an excipient, a binder, etc. This sweetener having aspartame entrapped with CD shows a higher dispersibility and solubility of the

aspartame in water than one having no entrapped  
aspartame that is pulverized, granulated or pelletized  
from a solution or a slurry of a simple mixture of the  
aspartame and CD. Thus this preferred dried product  
5 according to the present invention not only has high  
dispersibility and solubility in water but also has  
enhanced stability against storage in water, so it can  
be used advantageously as a sweetener for incorporation  
in processed foods and beverages or as a table  
10 sweetener.

Another significant feature of the present  
invention is to provide fluid or semi-fluid aqueous foods  
wherein the aspartame keeps its initial quality for an  
extended period. The stability of the quality of the aspartam  
15 can be improved without entrapping it in the CD.  
Furthermore, the amount of the CD added can be held to  
a minimum, so an aqueous food that has a very low  
calorie content and which can be stored for an extended  
period can be produced with high efficiency according  
20 to the present invention. To give a specific example,  
a beverage as sweet as an ordinary soft drink, for  
example, one containing 5 g of sucrose per 100 ml, can  
be offered by the present invention while reducing the  
calorie content to a negligible level, i.e., about  
25 1/100 to 5/1,000 of the calorie content of the  
conventional soft drink.

The present invention also includes compositions which contain, either alone or in combination with a cyclodextrin as described above, a sucrose fatty acid ester. Preferred aspects of this invention therefore  
5 relate to an aspartame-containing aqueous food that contains 0.008 to 3 wt% of a sucrose fatty acid ester.

The term "sucrose fatty acid ester" as used in the present invention refers to an edible surfactant composed of sucrose and a fatty acid, many examples of  
10 which are known to the prior art for use in other methods and compositions. Numerous references are available which describe sucrose fatty acid esters and their production. These include "Sucrose Ester of Fatty Acid; Part 1", Ishizuka et al., Journal of Food  
15 Industry, 14 (1971); "Sucrose Ester of Fatty Acid; Part 2: Fundamental Physiological Properties", Watanabe et al., Journal of Food Industry, 14 (12), 65-71 (1971); "Sucrose Ester of Fatty Acid; Part 3", Watanabe, Journal of Food Industry, 14 (16), 105-111 (1971); "The  
20 Surface Chemistry of Alkyl Esters of Sucrose", Osipow et al., J.A.U.C.S., 35, 127-129 (1958); and "DK-Ester" (a monograph on sucrose fatty acid ester by DKS International, Inc.).

Since sucrose fatty acid esters  
25 are digested in the human body and absorbed as sucrose along with a fatty acid, their use in foods is unlimited. The exact nature of these compounds varies

greatly according to the proportions of the fatty acids  
(e.g., stearic acid, palmitic acid, lauric acid and  
oleic acid) used to form the ester. Any type can be  
used in the present invention and preferably it has  
5 an HLB value within the range of from 2 to 19. The  
sucrose fatty acid ester is preferably used in a  
concentration of 0.008 to 3 wt% of the food in which it  
is incorporated. If the concentration of the sucrose  
fatty acid ester is less than 0.008 wt%, its intended  
10 ability to stabilize aspartame is not achieved. There  
is no particular upper limit to the concentration of  
the sucrose fatty acid ester, but, organoleptically, it  
should not exceed 3% (in terms of the concentration in  
the actual form of food intake; this also applies to a  
15 concentrated food). More specifically, the range of  
0.01 to 1 wt% is optimum for enhancing the stability of  
aspartame in water without impairing the organoleptic  
characteristics of the food. As long as the concentra-  
tion of the sucrose fatty acid ester is within the  
20 range of 0.008 to 3 wt%, any other edible surfactant  
such as glycerol fatty acid ester, sorbitan fatty acid  
ester or soybean phospholipid may also be used in a  
concentration of about 0.005 to 1%.

The sucrose fatty acid ester may be added by any  
25 method and at any time during preparation of the  
composition so long as it is uniformly dispersed or  
dissolved in the aqueous phase of the final product.

However, if the aspartame is heated in the presence of water during food production or if there is the possibility of reduced aspartame stability due to the aging, it is more effective to add

5 the sucrose fatty acid ester prior to or simultaneously with the aspartame. It is also desirable to incorporate either cyclodextrin as described above or a conventional agent capable of enhancing the stability of aspartame in water or to incorporate an agent to

10 control the pH to be in a stable region for aspartame (e.g., a pH lowering agent such as an organic acid or amino acid, a pH increasing agent, or a pH buffering agent).

The compositions of the invention are suitable for

15 use in any aqueous food to replace sugars normally used as sweeteners. The term "aqueous foods" as used herein refers to all foods except dried foods and oily foods, and includes beverages such as juice, cola, sports drinks (i.e., isotonically balanced drinks), coffee,

20 tea, cacao dairy milk, fruit-flavored milk, coffee-flavored milk, soybean milk, conditioned soybean milk, flavored soybean milk, amazake (sweet sake made from fermented rice), silko (azuki soup with rice cake), ginger ale, milk shakes, tomato juice and other

25 vegetable juices; yogurt, jelly, Bavarian cream, pudding, and mousse; sauces such as ketchup, mayonnaise, salad dressings, Worcester sauce, fruit-

flavored sauce, chocolate sauce, tomato sauce and chili  
sauce; creams, toppings, fillings and jams; frozen  
desserts such as ice creams and sherbets; pickle  
syrops, and pickling syrup; chewing gum, bean jellies,  
5 condensed milk, cooked beans and yokan (Japanese sweet  
jelly of bean). The present invention is particularly  
effective for enhancing the stability of aspartame in  
the above-named aqueous foods and beverages which are  
canned, bottled, pouched, packed or otherwise packed in  
10 manners suitable for shipping and display at room  
temperature or in a chilled state.

A particularly preferred embodiment of the  
invention comprises fluid and semi-fluid aqueous foods  
produced as described herein. The term "fluid aqueous  
15 foods" as used herein refers to beverages such as those  
described above or other readily flowing compositions  
of high water content. The term "semi-fluid aqueous  
foods" as used herein refers to viscous flowable foods,  
optionally containing solids, such as yogurt, jelly,  
20 Bavarian cream, pudding, ketchup, mayonnaise, salad  
dressings, chocolate sauce, tomato sauce, and similar  
items. The invention is of particular relevance to  
these fluid and semi-fluid aqueous foods because of the  
high water content of these foods, which normally  
25 contributes to rapid degradation of aspartame.

There is no particular limitation on the  
concentration of aspartame in these foods, and the

concentration may be properly determined according to the sweetness of the aqueous food in which the aspartame is to be used. Needless to say, the aspartame may be used together with known natural  
 5 sweeteners such as sucrose, saccharides, sugar alcohol and stevioside, as well as with artificial sweeteners such as saccharin.

The invention now being generally described, the same will be better understood by reference to certain  
 10 specific examples which are included herein for purposes of illustration only and are not intended to be limiting of the invention or any embodiment thereof, unless specified. In the examples, all percentages and parts are by weight.

15

Example 1

Aqueous solutions containing aspartame and CD in the amounts indicated in Table 1 were prepared. As a control, an aqueous solution containing only aspartame  
 20 was prepared. All samples were stored at 24°C to check the long-term stability of aspartame.

Table 1

	Sample composition		Percent residual aspartame	
	conc. of aspartame (mole/1,000 ml)	conc. of $\beta$ -CD (mole/1,000 ml)	stored at 24°C	
			18 days	45 days
25 1	0.365	0	92.8	80.0



2	0.365	0.002	93.5	81.8
3	0.365	0.012	94.0	83.0
4	0.365	0.023	97.9	87.1
5	0.365	0.083	99.0	88.6

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5           The table shows that the long-term stability of  
the aspartame could be improved by using  $\beta$ -CD in a low  
concentration which was only about five thousandths of  
a mole with respect to one mole of the aspartame. The  
aspartame mixed with  $\beta$ -CD in a molar ratio of about 1:5  
10 underwent little decomposition when it was stored at  
24°C for 18 days.

#### Example 2

"Cyclo TC" is the trade name for a cyclodextrin  
manufactured by Toyo Jozo Co., Ltd., with a cyclodextrin  
15 formulation of 30%  $\alpha$ -CD and 50% total CD. Cyclo TC (30  
parts) was mixed with water (90 parts) to form a  
slurry, to which was added a wet cake prepared by  
mixing aspartame (100 parts) with water (30 parts).  
While retained at 35-45°C, the mixture was agitated in  
20 a kneader for one hour. The resulting hydrous mixture  
was vacuum dried at 40-50°C and ground with a speed  
mill to produce a particulate compound wherein the  
aspartame was entrapped in Cyclo TC (water content:  
3.5%, particle size: 330  $\mu$ m in 50%).

25           The solubility of the so prepared sample of the  
present invention was compared with that of a control

sample which consisted of only bulk aspartame. The results are shown in Table 2 from which one can see that the dispersibility of the aspartame in water can be increased by entrapping it in the cyclodextrin, as  
 5 evidenced by a shorter time required for dissolving the sample of the present invention in water (1/3 to 1/5 of the time necessary for dissolving the control sample).

Table 2

	Dispersibility	Dissolution rate <sup>1)</sup>
10 sample of the present invention	dispersed and suspended in water as soon as the sample was put in water	4 min.
control sample	floating lumps prevented rapid dispersion in water	15 - 20 min.

- 15 1) The time until 1.0 g of aspartame completely dissolved in 100 ml of water (35°C) in a 200-ml beaker by stirring with a magnetic stirrer (100 r.p.m.) was measured.

Example 3

"Celdex N" is the trade name for a  $\beta$ -cyclodextrin  
 20 manufactured by Japan Maize Products Co., Ltd. Celdex N (100 parts) was mixed with water (200 parts) to form a slurry, to which was added a wet cake prepared by mixing aspartame (200 parts) with water (60 parts). The mixture was processed as in Example 2 to prepare a  
 25 particulate compound wherein the aspartame was entrapped in CD.

The solubility of the so prepared sample of the present invention was compared with that of a control sample which consisted of only bulk aspartame. The results are shown in Table 3, from which one can see that the water solubility of the aspartame entrapped in CD was higher than that of the untrapped aspartame by about 24% (at 5°C) and 10% (at 25°C).

Table 3

	Aspartame solubility (g/100 g H <sub>2</sub> O)	
	5°C	25°C
sample of the present invention	0.92	1.29
control sample	0.74	1.17

Example 4

Two parts of the particulate compound prepared in Example 3 was mixed with 98 parts of sucrose and 12 parts of water (binder), and a granular sweetener was prepared from this mixture by the fluid granulation process. Control No. 1 was prepared from a mixture of aspartame (1.3 parts), Celdex N (0.7 part) and sucrose (98 parts) by the same method. Control No. 2 consisting of only bulk aspartame was also prepared. The dispersibility, dissolution rate, and aspartame stability against storage (at 24°C) were checked for each sample. The results are shown in Table 4.

Table 4

	Solubility		Storage stability <sup>2)</sup> percent residual aspartame
	dispersibility	dissolution rate <sup>1)</sup>	
5	sample of the present invention	dispersed as soon as the sample was put in water 45 sec	96.0
10	control No. 1	dispersed as soon as the sample was put in water 150 sec	91.7
15	control No. 2	floating lumps pre- vented rapid dispersion in water 15 - 20 min	80.0

1) : The rate of dissolving 1.0 g of aspartame in 100 ml of water at 35°C as in Example 1.

2) : A solution containing 0.5 g of aspartame in 100 ml of water was stored in an incubator (24°C) for 45 days. Thereafter, the residual aspartame was determined by an analyzer of Hitachi, Ltd.

As Table 4 shows, the granular sweetener of the present invention could be dissolved in water in a period which was only 1/3 to 1/26 of the time necessary for dissolving the controls wherein the aspartame was not entrapped in CD; furthermore, the storage stability of the sample of the present invention was 5 to 20% higher than that of the controls.

Example 5

Water (75 parts) was added to Cyclo TC (30 parts), and the mixture was heated at 40°C. Then, a slurry prepared by adding aspartame (30 parts) to water (30°C) was added to the mixture. The resulting mixture was kneaded as in Example 1 to prepare a paste, which was dissolved under stirring in a mixture of a strawberry flavor (0.15 part), citric acid (0.3 part) and water (35 part) to provide a strawberry syrup (pH 4.2).

The syrup was stored in an incubator (15°C) for 60 days. The decomposition of the aspartame was negligible and its residual content was as high as 99.5%. In an organoleptic test, the number of the panelists who liked the flavor of a sample of the present invention was significantly larger than that of those who liked the flavor of a strawberry syrup having the same degree of sweetness.

Example 6

To 500 ml of Coca-Cola (manufactured by Fuji Coca-Cola Bottling Co., Ltd.), 0.35 g of the aspartame-entrapped compound prepared in Example 2 was added. The mixture was rebottled and sterilized by heating in water (80°C) for 20 minutes to prepare an aspartame-containing Coca-Cola. A control Coca-Cola containing 0.27 g of bulk aspartame was prepared by the same method.

The two Coke samples were stored in an incubator (24°C) for 45 days. The sample having the aspartame entrapped in CD retained 87.2% of the residual aspartame, which was about 10% higher than the percent residual aspartame in the control (79.8%). In an organoleptic test, the Coke sample having the aspartame entrapped in CD was found to have a more intense sweetness of better quality than the control.

#### Example 7

10 To 500 ml of commercial orange juice (manufactured by Pocca Lemon Co., Ltd.), 0.53 g of the aspartame-entrapped compound prepared in Example 1 was added. The mixture was bottled and sterilized by heating in water (80°C) for 20 minutes to prepare an aspartame-  
15 containing orange juice. A control orange juice containing 0.41 g of bulk aspartame was prepared by the same method.

The two orange juice samples were stored in an incubator (24°C) for 45 days. The sample having the  
20 aspartame entrapped in CD retained 86.3% of the residual aspartame, which was about 8% higher than the percent residual aspartame in the control (79.2%).

In an organoleptic test, the orange juice sample having the aspartame entrapped in CD was found to have  
25 a more intense sweetness of better quality and was more flavorful than the control.

Example 8

A solution of 1% of sucrose fatty acid ester (product of Dai-Ichi Kogyo Seiyaku Co., Ltd.) in 0.93% aqueous aspartame was put in 150 ml bottles, and heated at 80°C for 30 minutes. The bottles were then held in an incubator (44°C) for defined periods to determine the long-term stability of the aspartame. As controls, aqueous aspartame solutions containing no sucrose fatty acid ester were prepared. The solutions in both the test group and the control group had a pH of 6.2. The time-dependent change in the residual aspartame is shown in FIGURE 1, from which one can see that in the test group containing 1% of the sucrose fatty acid ester, 70% of the aspartame remained intact even after 30 days, but in the control group, almost all aspartame was lost in 5 days.

Example 9

A solution of 3% of sucrose fatty acid ester (product of Dai-Ichi Kogyo Seiyaku Co., Ltd.) in 0.82% aqueous aspartame was distributed among 150-ml bottles, and heated at 80°C for 30 minutes. The bottles were then held in an incubator (44°C) for defined periods to check the long-term stability of aspartame. As controls, aqueous aspartame solutions containing no sucrose fatty acid ester were prepared. The solutions

in both the test group and the control group had a pH of 6.5. The time-dependent change in the residual aspartame is shown in FIGURE 2, from which one can see that in the test group containing 3% of the sucrose fatty acid ester, at least 40% of the aspartame remained intact even after 30 days, but in the control group, almost all aspartame was lost in 2.5 days.

Example 10

10	Coffee powder (manufactured by Ajinomoto General Foods Inc.)	15 g
	Aspartame	1 g
	Marime® (creaming powder by Ajinomoto General Foods Inc.)	25 g
15	Sucrose fatty acid ester (DK-160 manufactured by Dai-Ichi Kogyo Seiyaku Co., Ltd.)	0 - 15 g
	Water to make	1,500 g

A mixture of the above indicated components was packed in 250-ml cans (tuna No. 4), sterilized by autoclaving (Fo value: 20) and cooled to make canned coffee preparations having a pH between 6.4 and 6.5. The preparations were checked for any decomposition of the aspartame that might have occurred during their production. They were also stored at room temperature for 3 months to examine the stability of the aspartame. The results are shown in Table 5 below.



Table 5

Test group	conc. of sucrose fatty acid ester (%)	conc. of aspartame		conc. of aspartame after 30 day storage at 24°C (%)
		before autoclaving (%)	after autoclaving (%)	
5				
1	0	0.067	0.044	0
2	0.1	0.067	0.050	0.035
3	0.3	0.067	0.065	0.045
4	1.0	0.067	0.063	0.054

10           The data shows that the decomposition of aspartame during heating and storage could be inhibited by adding the sucrose fatty acid ester to canned coffee preparations. The effectiveness of the fatty acid ester was particularly significant during storage.

15

Example 11

A mixture of aspartame (0.6 g), citric acid (0.25 g), 85% phosphoric acid (0.3 g), caramel (4 g), sodium citrate (0.1 g) and distilled water (150 ml) was heated to form a solution. To the solution was added 0.1 g of green leaves extract that had been dissolved in 25 ml of distilled water by heating. The resulting mixture was cooled in ice water and blended with 1 ml of a flavor mix consisting of 0.5 mg of cola essence W4723 (by Takasago Perfumery Co., Ltd.), 0.23 ml of vanilla extract and 0.25 ml of cola nut extract. To the

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mixture, 800 ml of cold carbonated water was added to make a total volume of 1,000 ml. The resulting solution was equally distributed among four bottles. During the preparation of these colas, samples of

5 sucrose fatty acid ester (product by Dai-Ichi Kogyo Seiyaku Co., Ltd.) were added in concentrations of 0.01%, 0.05%, 0.10% and 1.00%. The long-term stability of aspartame in the test cola preparations was compared with that of controls having no sucrose fatty acid

10 ester. The results are shown in Table 6 below, from which one can see that the stability of the aspartame in cola was improved by adding the sucrose fatty acid ester, and its effectiveness was increased as more sucrose fatty acid ester was used.

Table 6

5	amount of sucrose fatty acid ester added (%)	conc. and residual cont. of aspartame	time-dependent change in residual aspartame (at 24°C)			
			0 day	30 days	70 days	120 days
	0	conc. (mg%)	55.5	47.0	37.1	28.5
		residual cont. (%)	(100.0)	(84.6)	(66.9)	(51.4)
10	0.01	conc. (mg%)	55.5	49.4	40.6	34.5
		residual cont. (%)	(100.0)	(89.0)	(73.2)	(62.2)
15	0.05	conc. (mg%)	55.4	49.8	43.2	38.8
		residual cont. (%)	(100.0)	(89.9)	(78.0)	(70.1)
20	0.10	conc. (mg%)	55.3	50.1	46.6	41.8
		residual cont. (%)	(100.0)	(90.6)	(84.3)	(75.6)
25	1.00	conc. (mg%)	55.3	52.9	48.0	43.1
		residual cont. (%)	(100.0)	(95.6)	(86.8)	(77.9)

Example 12

One thousand grams of dairy milk was cooked at 80-85°C for 10 minutes and concentrated to 40% of the original volume by a vacuum concentrator (under indirect heating at 46-60°C). To 400 g of the concentrate, 1.7 g of aspartame and 330 g of SE 100 (a reduced starch hydrolyzate of Matsutani Kagaku Kogyo

Kaisha Ltd.) were added, and the mixture was cooled to cause the crystallization of lactose at a controlled rate. The cooled mixture was packed in cans.

During the preparation of these sugared condensed whole milk samples, a sucrose fatty acid ester was added simultaneously with the addition of the aspartame so that the final concentrations of the ester in the final product were 0.5%, 1.0% and 3.0%, respectively. The long-term stability of aspartame in the test samples were compared with that of controls having no sucrose fatty acid ester. The results are shown in Table 7.

Table 7

15	Sucrose fatty acid ester	Time-dependent change in percent residual aspartame (%)			
		0 day	30 days	70 days	120 days
	0% (control group)	100%	82.3%	50.1%	42.0%
20	0.5	100	87.0	71.4	66.6
	1.0	100	100.0	92.3	83.3
	3.0	100	100.0	97.8	91.6

The above data shows that the stability of the aspartame in condensed milk was greatly improved by adding the sucrose fatty acid ester, and its

effectiveness was increased as more fatty acid ester was used.

Example 13

A measured amount (250 g) of the pulp of mandarin oranges was prepared by a conventional method and packed in each of six No. 5 cans, which were also filled with 67 g of syrup samples having the formulations indicated below. The cans were sealed with an automatic vacuum curler and the contents were sterilized at 80-85°C (center temperature: 72-73°C) for 12 minutes and cooled.

Syrup formulations

	aspartame	water	coupling sugar*	sucrose fatty acid ester
control group	0.24 (g)	35 (g)	32 (g)	0 (g)
test group 1	0.24	35	32	0.03
test group 2	0.24	35	32	0.06

\* coupling sugar: a product manufactured by Hayashibara Seibutsu Kagaku Kenkyujo (Biological Institute)

Each of the three groups of canned syruped mandarin samples was divided into two groups, one of which was placed in a constant temperature bath at 24°C, and the other group was put in a bath at 44°C. They were stored in the respective baths for 3 months

to check the long-term stability of the aspartame. The results are shown in Table 8, from which one can see that the stability of aspartame could be greatly improved by adding the sucrose fatty acid ester. The effectiveness of the ester was enhanced as its amount was increased, but in the storage at 40°C, the addition of 0.03% ester was sufficient to give a residual aspartame content at least twice the level achieved in the absence of the ester.

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Table 8

	amount of sucrose fatty ester in syrup	storage temp. (°C)	residual aspartame (%)		
			0 month	1 month	3 months
15 control group	0	24	100.0	70.5	61.4
		44	100.0	51.3	37.8
test group 1	0.03	24	100.0	100.0	96.5
		44	100.0	96.5	71.4
test group 2	0.05	24	100.0	100.0	98.6
		44	100.0	100.0	90.5

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Example 14

Sucrose fatty acid ester (product of Dai-Ichi Kogyo Seiyaku Co., Ltd.; 30 g) was melted under heating at a temperature not higher than 65°C, 5 g of aspartame was admixed with the molten sucrose fatty acid ester, and the admixture was cooled and pulverized into the powdered premix.

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A 3.5 g portion of the premix was dissolved into 1000 ml of distilled water, and the solution was adjusted to pH 3.0 by an addition of citric acid solution (concentration of 20%) and packed in bottles.

5        The bottled solution was heated at 80°C for 30 minutes and placed in a constant temperature bath at 44°C for 30 days for the purpose of measuring storage stability of aspartame.

10       As a control, an aspartame solution prepared by dissolving 0.5 g of aspartame into 1000 ml of distilled water was used.

15       The results of the time-dependent change in percent residual aspartame content are shown in Table 9, from which one can see that the storage stability of aspartame could be greatly improved by the addition of sucrose fatty acid ester over the control. That is, the sample containing the sucrose fatty acid ester retained more than 90% of the residual aspartame and a loss of sweetness could not be perceived for the sample  
20       in an organoleptic test, while the control, for which a loss of sweetness could clearly be perceived in the test, retained about 40% of the residual aspartame. In addition, the sample of the invention showed a great improvement in solubility and wettability of aspartame  
25       over the control.

Table 9

		conc. of aspartame residual cont. of (aspartame)	time-dependent change in residual aspartame (at 44°C)	
			0 day	30 days
5	test group	conc. mg/dl residual (cont.)	50.5 (100)	46.5 (92.0)
	control group	conc. mg/dl residual (cont.)	50.3 (100)	21.3 (42.3)
10				

The invention now being fully described, it will be apparent to one of ordinary skill in the art that many changes and modifications can be made thereto without departing from the spirit or scope of the invention as set forth herein.



CLAIMS :

1. A stabilized aspartame composition comprising aspartame and a sugar derivative selected from the group consisting of a cyclodextrin and/or a sucrose fatty acid ester.  
5
2. The composition of Claim 1, wherein said sugar derivative is a cyclodextrin and the molar ratio of aspartame to cyclodextrin is from 200 : 1 to 1 : 50, preferably from 100 : 1 to 1 : 50.
- 10 3. The composition of Claim 1 or 2, wherein part of all of the aspartame is entrapped in the cyclodextrin.
4. The composition of any of the Claims 1 to 3, wherein the cyclodextrin is  $\alpha$ -cyclodextrin,  $\beta$ -cyclodextrin,  $\gamma$ -cyclodextrin, or a mixture thereof.
- 15 5. The composition of Claim 1, wherein said ester comprises a sucrose stearate, sucrose palmitate, sucrose laurate or a sucrose oleate.
6. The composition of Claim 1, wherein said sucrose fatty acid ester comprises 0.008 - 3 % by weight  
20 of said food.
7. A process for producing an aspartame sweetener composition, which comprises :  
mixing aspartame with a cyclodextrin in the presence of water or an aqueous solvent.

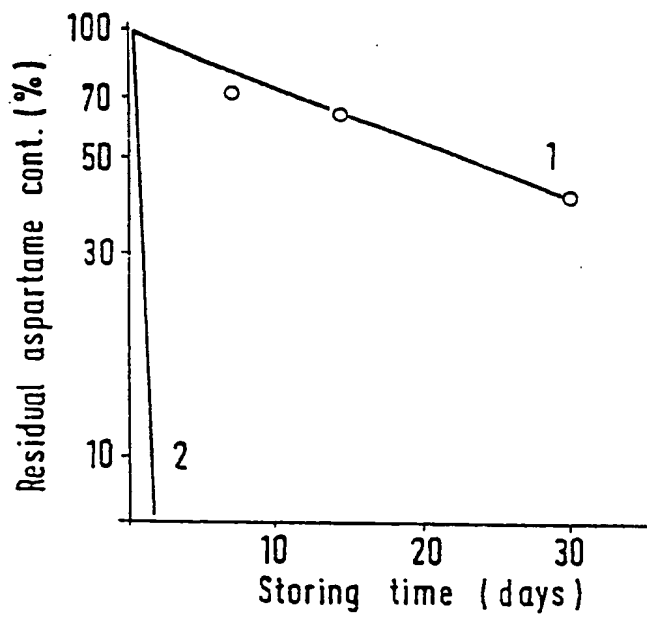
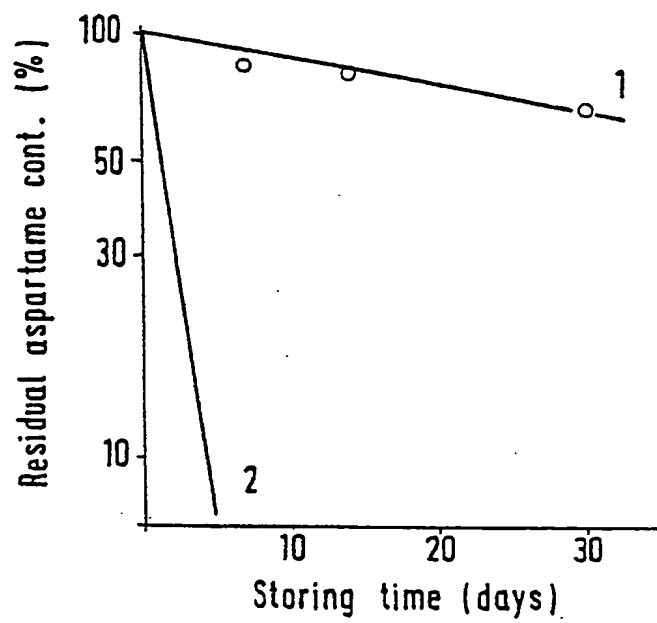
8. The process of Claim 7, wherein said process comprises the additional step of drying wherein a dried mixture of aspartame and cyclodextrin is produced.

9. The process of Claim 8, which comprises the additional step of shaping the dried mixture, optionally in the presence of an excipient or binder.

10. The process of Claim 7, wherein said mixing produces a slurry..

11. The process of Claim 7, which comprises :  
10 heating a solution or slurry of cyclodextrin in water at 30 - 90°C whereby a cyclodextrin composition is produced,  
mixing the cyclodextrin composition intimately with aspartame whereby an aspartame composition is  
15 obtained, and  
drying the aspartame composition at a temperature not higher than 80°C.

12. Aqueous food, especially fluid or semifluid food, containing a stabilized aspartame composition  
20 according to any of the claims 1 to 6:





European Patent  
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# EUROPEAN SEARCH REPORT

0097950

Application number

EP 83 10 6262

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl. 3)
A	DE-A-2 438 317 (GENERAL FOODS CORP.) * Pages 10, 11; example 1; claims 1, 6 *		A 23 L 1/236
A	EP-A-0 053 844 (AJINOMOTO CO) * Claims 1, 2, 4, 5 *		
A	Patent Abstracts of Japan Vol. 5, no. 4, 13 January 1981 & JP-A-55-131364		
			TECHNICAL FIELDS SEARCHED (Int. Cl. 3)
			A 23 L 1/00
The present search report has been drawn up for all claims			
Place of search BERLIN		Date of completion of the search 29-08-1983	Examiner SCHULTZE D
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